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I also certify that by virtue of an assignment registered under the Patents Act 1977, the application is now proceeding in the name as substituted.

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Signed Andrew Gersey

Dated 3 November 1999

GB9823080.8

By virtue of a direction given under Section 30 of the Patents Act 1977, the application is proceeding in the name of

DEVRO PLC
Modiesburn
Chryston
GLASGOW
G69 0JE
United Kingdom

Incorporated in the United Kingdom

[ADP No. 07054794001]

FRAUNHOFER-GESELLSCHAFT ZUR FÖRDERUNG DER ANGEWANDTEN FORSCHUNG E.V.
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Federal Republic of Germany

[ADP No. 07417058002]

Patents Form 1/77

Patents Act 1977
(Rule 16)

The Patent Office

1/77

220CT98 E399204-1 C25070
P01/7700 0.00 - 9823080.8

Request for grant of a patent

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The Patent Office

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THE PATENT OFFICE F 21 OCT 1998 RECEIVED BY HAND

1. Your reference — DCM/ED/P08466GB

2. Patent application number *(The Patent Office will fill in this part)* — 21 OCT 1998 — 9823080.8

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

Devro plc
Moodiesburn
Chryston
Glasgow G69 0JE

SECTION 30 (1977 ACT) APPLICATION FILED 16.4.99

Patents ADP number *(if you know it)* — 07547940001

If the applicant is a corporate body, give the country/state of its incorporation — Scotland, United Kingdom

4. Title of the invention — Extrusion of Cellulose Film

5. Name of your agent *(if you have one)*

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

Cruikshank & Fairweather
19 Royal Exchange Square
Glasgow G1 3AE

Patents ADP number *(if you know it)* — 547002

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number

Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application	Date of filing *(day / month / year)*

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'Yes' if:*

a) any applicant named in part 3 is not an inventor, or
b) there is an inventor who is not named as an applicant, or
c) any named applicant is a corporate body.
See note (d))

Yes

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9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

	Continuation sheets of this form
Description	9
Claim(s)	1
Abstract	Nil
Drawing(s)	2

10. If you are also filing any of the following, state how many against each item.

Priority documents	
Translations of priority documents	
Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*	
Request for preliminary examination and search *(Patents Form 9/77)*	
Request for substantive examination *(Patents Form 10/77)*	
Any other documents *(please specify)*	

11. I/We request the grant of a patent on the basis of this application.

Signature Cruikshank & Fairweather Date 20 October 1998

12. Name and daytime telephone number of person to contact in the United Kingdom

D C MacDougall
0141 221 5767

Warning

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Notes

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EXTRUSION OF CELLULOSE FILM

The present invention relates to an apparatus and process for the production of extruded cellulose film, particularly cellulose film (including sheets, tubes etc.) produced according to the well known amine-oxide extrusion process. Especially, it relates to the avoidance of variations in thickness and edge wrinkling.

The production of extruded cellulose articles, such as fibres, sheets or tubes has been known for many years. In this so called "viscose" process, cellulose is derivatised with carbon disulphide (CS_2) and solubilised in diluted sodium hydroxide (NaOH) to form a viscous solution, and the solution is extruded. The extruded cellulose is then regenerated and reverts to its solid form. The viscose process has been used for the manufacture of sausage casings, flat cellophane films, rayon fibres, bottle caps etc.

More recently, the so called "amine-oxide" process has been developed wherein the cellulose is dissolved in a mixture of water and an amine-oxide solvent. A commonly used amine-oxide solvent is the tertiary amine-oxide NMMO (N-methyl morpholine N-oxide). This solvent is able to dissolve cellulose without having to first derivatise the cellulose, as in the viscose process. Once solubilised the cellulose will precipitate from the solution as a regenerated cellulose product by contacting the solution

with a precipitation liquid which is a non-solvent for cellulose and a solvent for NMMO. The most frequently used precipitation liquid for the amine-oxide process is water.

In the extrusion step of the amine-oxide process when used to produce tubular casings (for use, for example in the food industry), the tube is extruded into a bath of precipitation liquid. Precipitation liquid is also maintained within the extruded tube so as to solidify the cellulose from the inside. A slight positive pressure of air may be maintained above the precipitation liquid within the extruded tube so as to expand the tube in the transverse direction (ie. transverse to the machine extrusion direction). The presence of precipitation liquid within the extruded cellulose tube causes process difficulties after the tube emerges from the precipitation bath, since it is necessary to cut the tube at regular intervals in order to allow the liquid to drain away. Failure to do so, leads to undesirable stretching of the tube and variations in diameter and thickness.

It is an object of the present invention to mitigate these problems.

The present invention provides an apparatus for the production of extruded cellulose film, which comprises

- extrusion means for continuously extruding a cellulose solution to form a cellulose film;
- precipitation means for solidifying the extruded cellulose film; and

- draw means positioned downstream of the extrusion means for continuously drawing the extruded cellulose film from the extrusion means.

Thus, it has surprisingly been found that providing a positive transport mechanism in the form of draw means to continuously draw the extruded film away from the extrusion means gives good control over the thickness of the extruded film.

The cellulose solution for extrusion may be a viscose solution or an amine-oxide solution, according to known technology.

The extrusion means generally comprise an extrusion die shaped to extrude a cellulose film of desired shape and dimensions. In particular, the film may be in the form of a flat sheet or may alternatively be in the form of a tube. Flat cellulose sheets are used in the food industry for wrapping food products. Cellulose tubes are used to encase food products, such as sausages, salami or other cased food products. As mentioned above, particular problems arise with the production of tubular cellulose products, since it is necessary to maintain a volume of precipitation liquid within the tube to assist solidification thereof.
According to a preferred feature of the present invention, when the extruded cellulose film is tubular the draw means is in the form of a pair nip rollers which draw the flattened tube away from the extrusion means. Preferably, the nip rolls act to hold the sides of the tube together to form a seal to retain the precipitation liquid in a fixed

volume defined between the extrusion means and the draw means. This avoids carry over of liquid downstream and thus minimizes the need to cut the extruded cellulose tube at regular intervals to release carried over liquid. This represents a saving in materials and also in labour costs.

In order to facilitate flattening of the tube prior to the draw means (eg. the nip rollers) it is preferred to provide collapsing means for collapsing the tube from its tubular form towards a flattened form. The collapsing means may be in the form of opposed non-parallel guide plates having a decreasing spacing towards the draw means, typically at an angle of 10 to 35°, preferably 15 to 25°.

In a particular embodiment, the draw means and the collapsing means are combined into a single unit in the form of a pair of opposed non-parallel moving belts which are spaced at an end where the extruded tube enters and come closer progressively towards a nip at the draw means. The moving belts have the benefit of positively transporting the extruded tube through the collapsing means and into the draw means.

Where the collapsing means are stationary, they will generally be coated with a low friction material, such as PVDF. Where the collapsing means are in the form of a moving belt, a belt such as a rubber belt having a suitable surface roughness for transporting the cellulose tube will be chosen.

It is found that the presence of the collapsing means helps to avoid wrinkling of the edges of the flattened tube.

The precipitation means generally comprises a liquid. The liquid may be present in a bath or may be showered onto the extruded film. In the case of the amine-oxide process, the precipitation liquid is generally water or dilute aqueous amine-oxide solution.

The draw means is generally in the form of a pair of nip rollers having a surface formed of a material having appropriate frictional qualities for gently gripping the extruded film without damaging it. The draw means may be driven at a speed which is slightly faster (for example 10 to 250% faster) than the speed of the extrusion means in order to expand the extruded cellulose film in the longitudinal machine direction.

The present invention also relates to a corresponding process for the production of extruded cellulose film.

The present invention when applied to the production of extruded cellulose tube allows the maintenance of a fixed internal volume of precipitation liquid downstream of the extrusion means and the prevention of liquid carry over. It also enables the production of uniform flat tube of a constant wall thickness.

Embodiments of the present invention will now be described by way of example only, with reference to the drawings wherein:

Figure 1 is a schematic view of a first embodiment of the invention for the production of cellulose tube and employing separate draw means and collapsing means;

Figure 2 is a schematic view of a second embodiment employing a combined draw means and collapsing means in the form of a pair of non-parallel moving belts; and

Figure 3 is a schematic diagram of the overall extrusion process.

Figure 1 shows schematically a first embodiment comprising an extrusion means 1 including an extrusion die in annular form for extruding a cellulose tubular film 2 into a bath of precipitation liquid 6 via an air gap. In the case of an amine-oxide process, the cellulose solution for extrusion is a solution of cellulose and amine-oxide in water and the precipitation liquid is water or dilute amine-oxide solution. The extruded cellulose tube passes downwardly through the precipitation liquid until it encounters a collapsing device 3 in the form of a pair of opposed non-parallel plates coated with a low friction polymer (eg. PVDF). Each plate is inclined at an angle J of approximately 20° to the longitudinally machine direction (shown in a chain-dotted line). The collapsing means progressively collapses the tube into a flat form. The flattened tube is then drawn into the nip of a pair of rollers 4 which squeeze together the opposite sides of the flattened tube so as to form an effective seal and to supply sufficient pressure to the tube to maintain the extruded tube under a slight tension.

The distance H between the nip of the rollers and the initiation of collapsing of the tube walls can be varied experimentally until optimum results are achieved. Generally, the distance is in the region 10 to 100 centimetres.

An amount 7 of precipitation liquid is also maintained inside the extruded cellulose tube by a means (shown in Figure 3) which maintains the level of liquid inside the tube approximately the same as that outside the tube and assists solidification of the interior of the extruded tube. An air space 8 is left above the precipitation liquid within the tube which is maintained at slightly above atmospheric pressure (eg. 0.1 to 10 mbar) to assist expansion of the extruded film in the transverse direction.

The effect of the nip rollers 4 is firstly to draw the extruded cellulose tube from the extrusion means thereby maintaining a slight tension in the tube which is found to minimise variations in thickness. Secondly, the nip rollers help define a closed volume of extrusion liquid 7 so that extrusion liquid is not carried further downstream with the cellulose film, which would otherwise necessitate cutting the film to release the liquid.

Figure 2 shows a second embodiment in which analogous parts are marked with the same reference numerals. It differs from the first embodiment in that the function of the collapsing means and the roller draw means is combined in a pair of opposed non-parallel moving belt conveyors 5. Each conveyor comprises a belt 9 which passes around a nip

roll 4 at one end and around an idler roll 10 of smaller diameter at the other end of its travel. The surface of the belts has an appropriate roughness for frictionally engaging and collapsing the tube walls before they are fed into the nip defined between the nip rollers 4. The internal run of each belt is inclined at an angle J of approximately 20° as before. The benefit of the moving belt conveyors is that the extruded cellulose film (which may still be somewhat delicate) is gently engaged by the moving conveyors and progressively fed into the nip of the rollers.

Figure 3 is an overall schematic diagram of the process and illustrates various processing conditions. Dope (ie. cellulose-amine-oxide aqueous solution) at approximately 100°C is stored in storage vessel 12. It is transported via extrusion device 14 and an extrusion screw through pump 16 to extruder 1 where the cellulose tube is extruded as described previously. A constant volume of water 7 is maintained in the space between the nip rolls 4 and the extruder by control means (not shown). Input pump 18 and inlet line 19 together with outlet pump 20 and outlet line 21 continuously flush water through the contained volume 7. Clean water is introduced and a dilute solution of water and NMMO (in the case of a typical amine-oxide process) is discharged.

Similarly, a constant volume of air is maintained in the space 8 via a line 22 at a slightly supra-atmospheric pressure in the range 0.1 to 10.0mbar (for example 1mbar)

to expand the tube transversely.

The extruded tube is collapsed and drawn down through nip rolls 4 as described previously and passes around a succession of guide rolls 24 within the water bath 6 to complete the cellulose solidification process. The flattened tube is then passed through a succession of water washing tanks (not shown) to remove any residual traces of amine-oxide; before plasticiser is applied to the outside of the tube and the tube is reeled for storage.

In this way, water is prevented by the nip rolls from being carried downstream, thus avoiding the need to cut the tube at regular intervals to release trapped water, as in prior art processes. The process allows flat cellulose tube of tightly-controlled dimensions to be produced.

Claims

1. Apparatus for the production of extruded cellulose film, which comprises

- extrusion means for continuously extruding a cellulose solution to produce a cellulose film;
- precipitation means for solidifying the extruded cellulose film; and
- draw means positioned downstream of the extrusion means for continuously drawing the extruded cellulose film from the extrusion means.

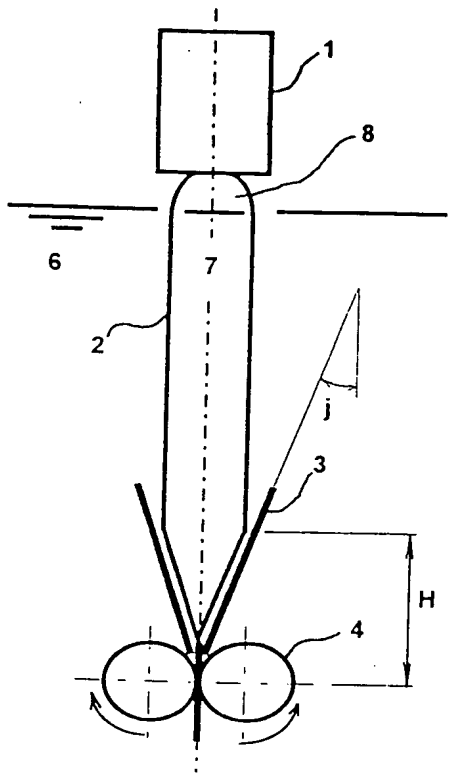


FIGURE 1 : PLATE SLIDE COLLAPSORS AND BOTTOM SQUEEZE ROLLS

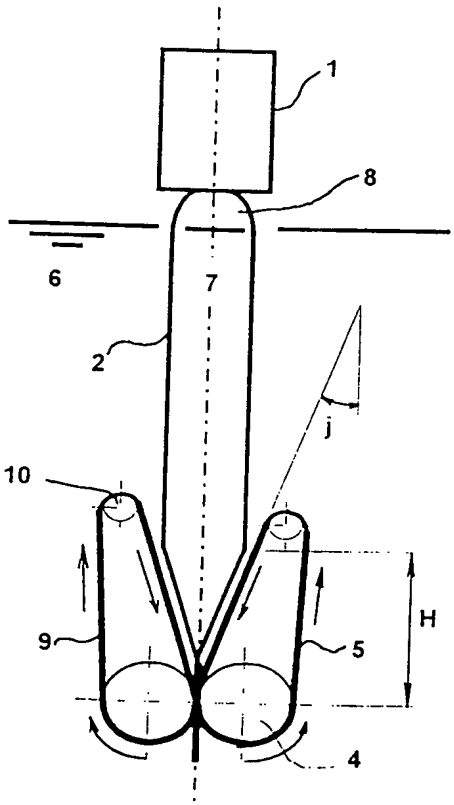
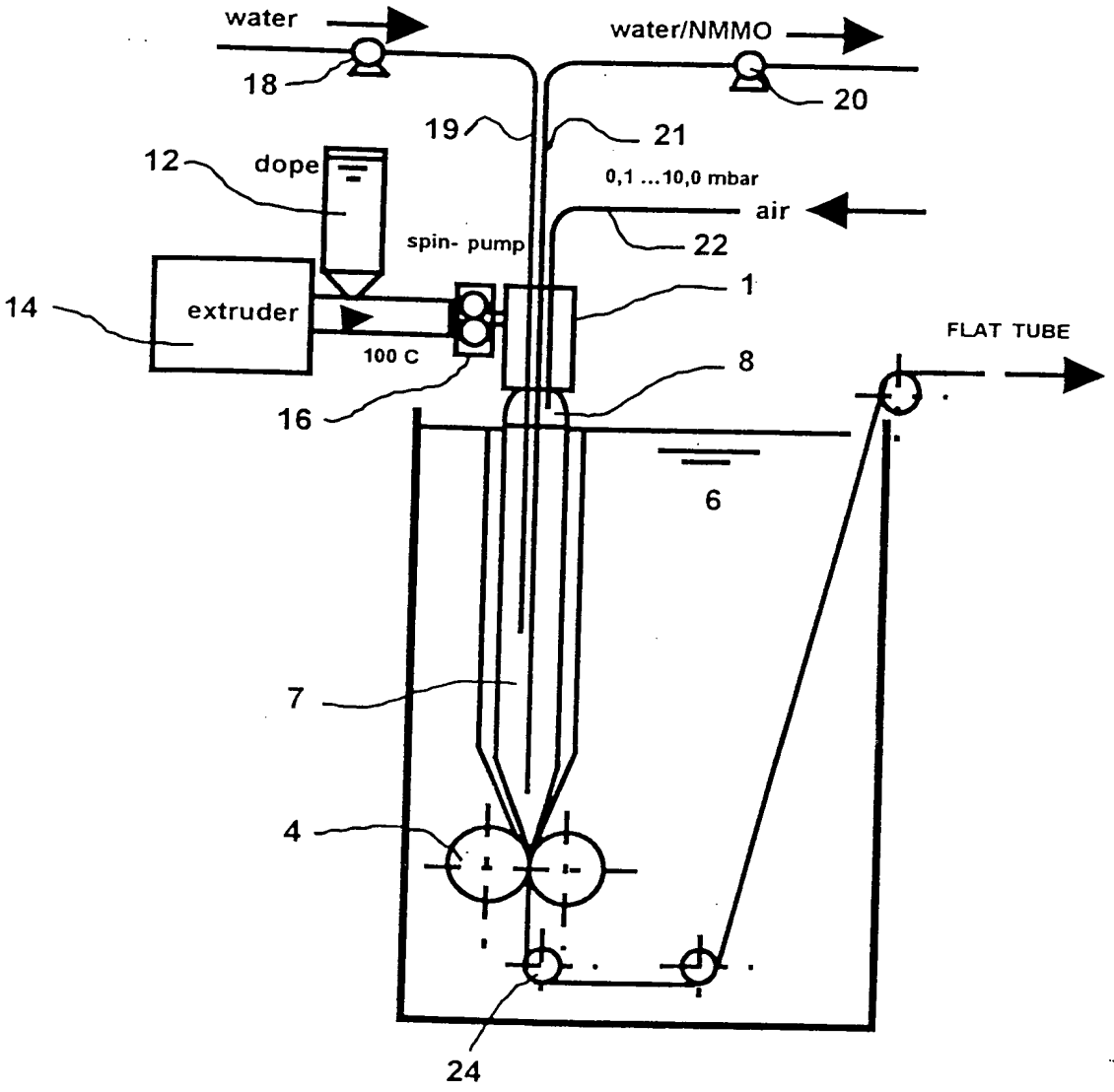


FIGURE 2 : MOVING BELT COLLAPSORS COMBINED WITH BOTTOM SQUEEZE ROLLS


water
water/NMMO
18
20
19
21
12
dope
0,1 ...10,0 mbar
air
spin- pump
22
1
14
extruder
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FLAT TUBE
8
16
6
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FIGURE 3 :

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